HIGHWAY RESEARCH REPORT

STATISTICAL ANALYSIS OF PORTLAND CEMENT TEST DATA

FINAL REPORT

68-12

STATE OF CALIFORNIA

TRANSPORTATION AGENCY

DEPARTMENT OF PUBLIC WORKS

DIVISION OF HIGHWAYS

MATERIALS AND RESEARCH DEPARTMENT

RESEARCH REPORT

NO. M & R 6-35149

Prepared in Cooperation with the U.S. Department of Transportation, Bureau of Public Roads August, 1968

STATE OF CALIFORNIA—TRANSPORTATION AGENCY

RONALD REAGAN, *Governor*

DEPARTMENT OF PUBLIC WORKS
DIVISION OF HIGHWAYS
MATERIALS AND RESEARCH DEPARTMENT
5900 FOLSOM BLVD., SACRAMENTO 95819

August, 1968

Final Report
M&R No. 635149
Federal No. F-4-13
Subproject 3916Y

Mr. J. A. Legarra
State Highway Engineer

Dear Sir:

Submitted herewith is a research report titled:

STATISTICAL ANALYSIS OF

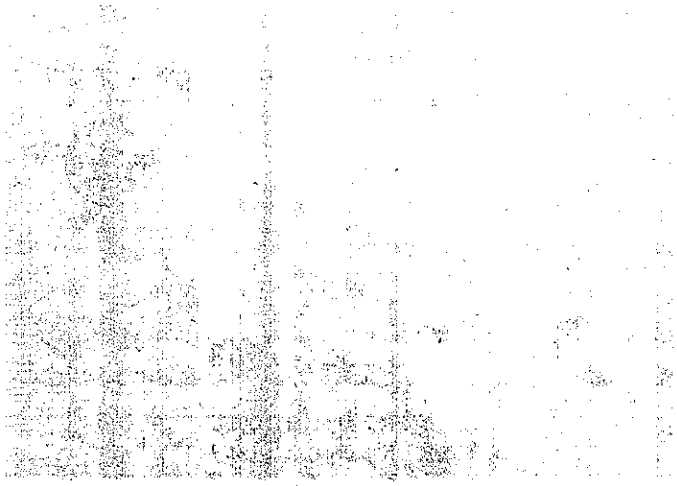
PORTLAND CEMENT TEST DATA

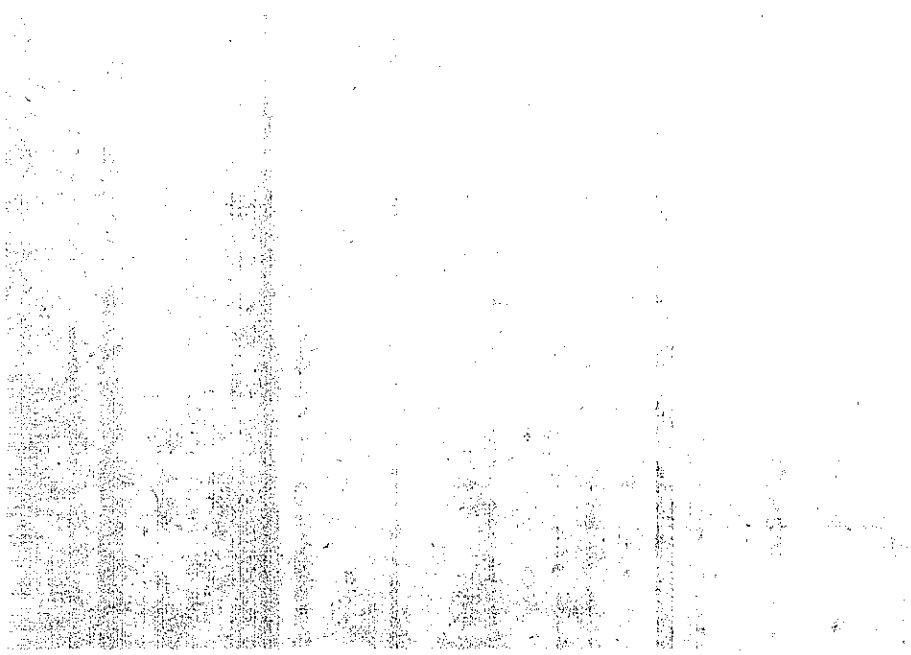
DONALD L. SPELLMAN
Principal Investigator

J. R. STOKER
ROBERT W. FORD
Co-Investigators

Very truly yours,

JOHN L. BEATON
Materials and Research Engineer





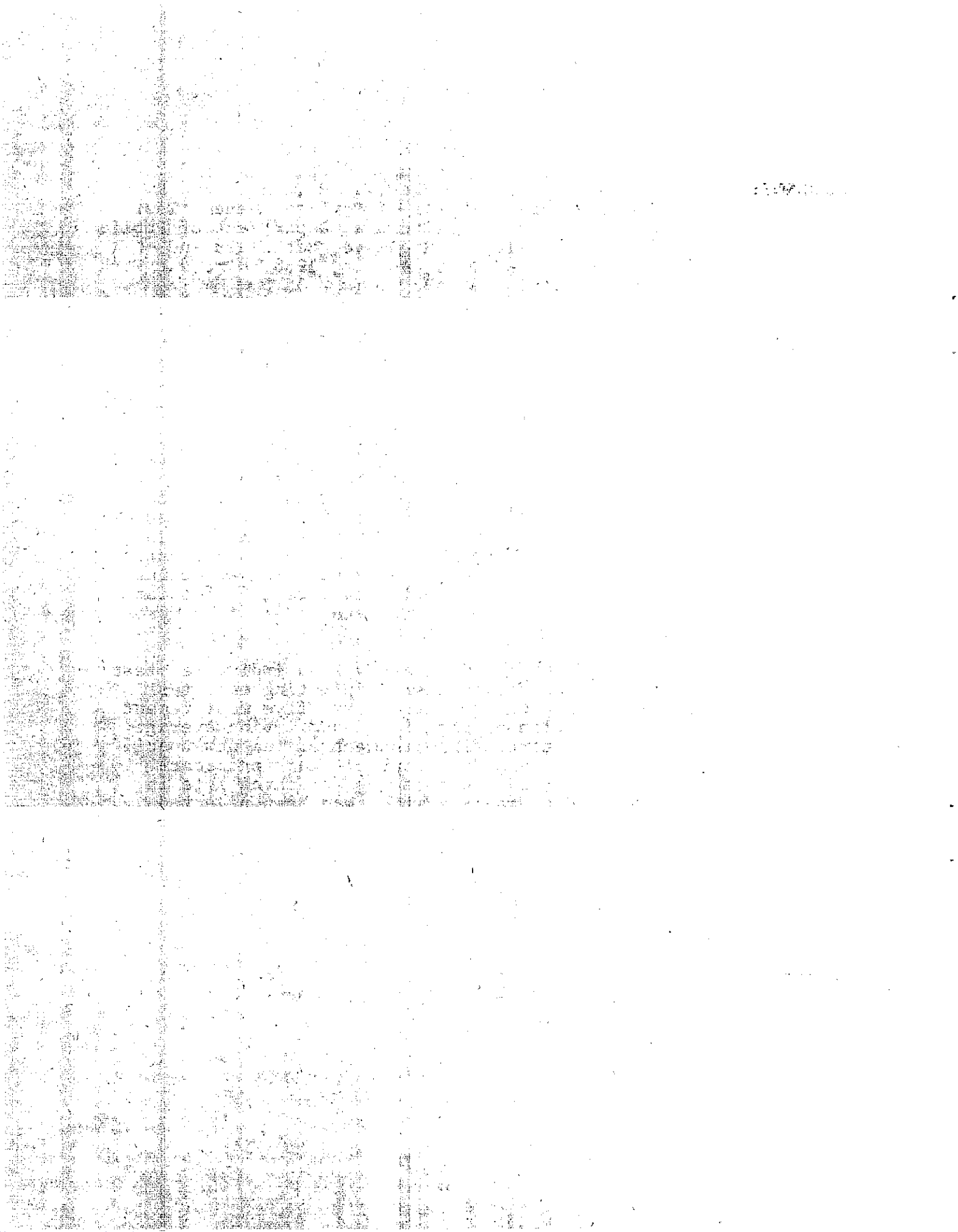
REFERENCE: Spellman, D. L., Stoker, J. R., and Ford, R. W. "Statistical Analysis of Portland Cement Test Data", State of California, Department of Public Works, Division of Highways, Materials and Research Department, August, 1968. Project Work Order No. M&R 635149.

ABSTRACT: This report contains proposals for specification limits on compressive strength, fineness, contraction in air, loss on ignition, and percent alkalies of Type II portland cement, for use on California Highway Projects.

Averages and standard deviations were determined for routine cement tests performed at the Materials and Research Department during the period from 1964 through 1966. Tentative specification limits were established which would permit rejecting "lots" of cement on the basis of being out of control rather than accepting cement which meets existing liberal specifications, but which may deviate widely from normal production.

During 1967, approximately 2800 individual test results were evaluated by plotting each test result and the average of the five most recent test results on individual and moving average control charts. Enforcement of the new specifications using the control chart procedure would have caused rejection of three lots in addition to the six which had been rejected under existing specifications. Producers of portland cement furnished to the California Division of Highways would not be affected by the new proposed specifications, unless they fail to maintain a good record of product quality control.

KEY WORDS: portland cements, properties, specifications, statistical analysis, data, control charts



ACKNOWLEDGMENT

This project was performed in cooperation with the U. S. Department of Transportation, Federal Highway Administration, Bureau of Public Roads, Agreement No. F-4-13.

The opinions, findings, and conclusions expressed in this report are those of the authors and are not necessarily those held by the Bureau of Public Roads.

STATISTICAL ANALYSIS OF PORTLAND CEMENT TEST DATA

INTRODUCTION AND BACKGROUND

Portland cement used in California highway construction is accepted by certification from the cement producer. Since the material is sampled at the jobsite and forwarded to the Materials and Research Department for testing, the cement is often used before acceptance testing is completed.

In general, the quality control exercised by cement producers has been excellent. On a few occasions however, cement has been used which had considerably different properties than those specified. Since the major portion of cement specifications now in use were developed a number of years ago when certain properties such as fineness, and early strength requirements were much lower, the off-quality cements have sometimes met specification requirements while causing considerable inconvenience because of sudden large deviations from normal. Table 1 illustrates how far some of the present ASTM limits miss the mark in controlling the properties of Type II cement.

This project was initiated to determine what limits could be set to permit accepting normal chance variations in measured properties of cement samples and rejecting samples showing variations of such magnitude as to indicate a lack of quality control. Control limits were to be based on the routine test results observed for samples tested during a period of at least a year.

CONCLUSIONS

1. Specifications now in use for Type II modified cement do not adequately control the properties of cements purchased by the California Division of Highways. Specification limits could be modified to more nearly coincide with the ranges of values encountered in routine testing of the product.

2. Control charts can be of help in controlling cement properties by providing guidelines for alerting producers to control problems, making decisions regarding acceptance of material, and determining when corrective action should be taken.

RECOMMENDATIONS

1. More realistic specification limits for the following properties of Type II modified cement should be adopted by the California Division of Highways:

   Compressive strength at 3 days and 7 days
   Fineness by air permeability method
   Contraction on drying (Test Method No. Calif. 527)
   Alkalies, as Na_2O
   Loss on Ignition

2. The specification limits outlined in this report should be discussed with cement producers as a step toward developing reasonable and enforceable specifications based on statistical concepts.

3. Data on other cement properties of interest, such as 28-day compressive strength and the properties of the less commonly used Types III and V cement should be accumulated and analyzed as a means of refining specifications.

4. The Materials and Research Department should alert portland cement producers to control problems whenever control charts for routine tests indicate that such problems exist.

5. The Materials and Research Department should continue to cooperate with each cement producer, on an individual basis, to conduct regularly scheduled cooperative tests on split samples of cement from current production. This procedure will enable each producer to determine the appropriate control values for his product.

PROCEDURE

Routine test data for each cement mill for a period of one year or more (usually the year 1965) were tallied. The average and standard deviation were calculated using the short Method No. 1 of ASTM Manual on Quality Control of Materials[1]. The average and standard deviation were calculated for the population of samples from all cement mills in California as well as for the samples from each mill. (See Tables 2 through 7.)

Tentative control limits for 3 and 7-day compressive strengths and for fineness by the air permeability method were calculated using the following equations:

$$\text{Specification Limits} = \bar{X} \pm 2.58\,\sigma \qquad (P = 0.01)$$

$$\text{Control Limits for Moving Average of 5} = \bar{X} \pm (2.58\,\sigma \div \sqrt{5}) = \bar{X} \pm 1.15\,\sigma$$

Where

$\bar{X}$ = Average) for the population of Materials
σ = Standard deviation) and Research Department routine test data for all brands of cement used by the California Division of Highways

(P = 0.01) indicates that chance variation will cause only 1% of all test results for a given property to occur outside the specified limits. Any test exceeding the limits should be investigated to determine an assignable cause for being "out of control"

Maximum control limits for alkalies, contraction, and ignition loss were calculated using the following equations:

$$\text{Specification Limits} = \bar{X} + 2.33\,\sigma \qquad (P = 0.01)$$

$$\text{Control Limit for Moving Average of 5 Tests} = \bar{X} + (2.33\,\sigma \div \sqrt{5}) = \bar{X} + 1.04\,\sigma$$

[1] Refers to the list of references at the end of text

The tentative control limits were adjusted where necessary in order to accept all tests which are "in control" from each mill. Since the control limits for alkalies, based upon the entire population of cement brands were found to be much higher than the control limits based on the data from any single cement mill, the present specification limit was retained and the moving average control limit was selected as that calculated for a brand of cement which has a representative standard deviation for alkalies and which conforms to the existing alkali specification at the 99% confidence level.

Control limits for loss on ignition, based on the data for 1964 through 1966, would have caused a large number of rejections if enforced during 1967 (an unusually wet year). The limits were therefore set at levels which permit acceptance of the material tested in 1967.

Test data obtained during 1967 were plotted on individual and moving average control charts in order to determine how many cement tests exceeded the tentative control limits. Comparisons between the existing and tentative specification limits and between corresponding rates of failure are shown in Table 8.

DISCUSSION ON THE USE OF CONTROL CHARTS

The procedure for use of the control charts is as set forth in Test Method No. Calif. 908-A. An individual test result outside the specification limit may be waived if the corresponding moving average is within limits. The manufacturer should be notified of such a result so that he may investigate possible causes of an abnormal test and take corrective action if necessary to restore control. The cement manufacturer should also be notified whenever the moving average goes out of specifications.

Comment:

Since test results reported by two laboratories for the same sample may differ somewhat, it is advisable for the Materials and Research Department and each cement manufacturer to conduct a split-sample testing program on a regular basis and/or to exchange samples and test results whenever the Materials and Research control charts indicate that a serious control problem exists. Such a continuing cooperative testing program was initiated by the Materials and Research Department in June, 1968.

DISCUSSION OF SPECIFICATIONS AND RATES OF FAILURE

Table 1 illustrates the inadequacies of the present specifications for controlling the properties of Type II cements. None of the cements tested at the Materials and Research Department exceeded (or even approached) the specification limits of four of the properties commonly evaluated - 3-day and 7-day compressive strengths, fineness, and loss on ignition. The arrival on a job of cement which departs from the range of expected values for one of these properties could cause difficulties in meeting design strengths, scheduling removal of forms, excessive bleeding, or shrinkage cracking.

Table 9 shows that during 1967, twelve test results exceeded the proposed individual specification limits for one of the cement properties evaluated. Three of these results could have been waived on the basis that the moving average limit was not exceeded, and the manufacturer could have been notified that the product appeared to be out of control. Other opportunities to warn manufacturers that their control procedures did not appear to be completely satisfactory occurred on 27 occasions when only moving average control limits were exceeded.

Under the proposed specifications, the minimum 3-day and 7-day compressive strengths would be increased and maximum limits would also be imposed. A shipment of cement having a 3-day compressive strength of 1590 psi would have been rejected. Under present specifications, a drop in strength to a considerably lower level would not be cause for rejection even though it might cause considerable problems to the engineer and contractor.

Only one mill had failures reported for high alkali content. The 15.8% (6 tests out of 38) failure rate under the existing specification would have remained the same under the proposed specification. Use of warnings when the moving average exceeded the control limit could have alerted the producer in time to avoid some of the results exceeding the specification limits.

Since January 1961, the California Division of Highways has had a specification limit for Type II cement of .048% maximum contraction on drying (Test Method No.

Calif. 527). Of 548 samples tested in 1967, a total of 15 samples from five mills exceeded this limit. The maximum rate of failure for any one mill under strict enforcement would have been 11% (4 tests out of 36). Since we have come to regard .005% as a reasonable tolerance for testing error and differences between laboratories, a single test result would not be considered cause for rejection unless it exceeded .053% contraction. (A history of tests exceeding .048% contraction has been sufficient cause for withdrawal of certification privileges, however.)

A proposed specification for contraction would raise the specification limit to .051% maximum and add a moving average control limit of .045%. If this proposed specification were used in 1967, two contraction tests would have exceeded the proposed specification limit; one of these results would have been waived. There would have been three occasions on which the cement manufacturer would have been warned about his control on contraction.

The present specification limit of 3.0% maximum loss on ignition appears to be unrealistic. During 1967, a year of above average rainfall when hydration of cement clinker in outside storage could be expected to be higher than normal, the highest ignition loss of portland cement tested at the Materials and Research Department was 2.2%. This single test result which exceeded the tentative specification limit of 2.0% would have been considered a failure. The manufacturer could have been warned however, that a sample shipped six weeks earlier had caused the moving average to exceed the control limit.

Table 8 indicates that application of the proposed specification limits using the control chart method would have increased the rejection rate for shipments of Type II modified portland cement only slightly, and would have afforded the Materials and Research Department a number of opportunities for communicating with the manufacturers about their quality control procedure.

REFERENCES

1. "ASTM Manual on Quality Control of Materials" Prepared by ASTM Committee E-11 on Quality Control of Materials, Special Technical Publication 15-C, January, 1951. Published by the American Society for Testing and Materials

2. "Determination of Specification Compliance Using Moving Averages and Control Charts", Tentative Method No. Calif. 908-B, Materials and Research Department, State of California, Department of Public Works, Division of Highways, December, 1967

3. Materials Manual, Testing and Control Procedures Vol. II, State of California, Department of Public Works, Division of Highways

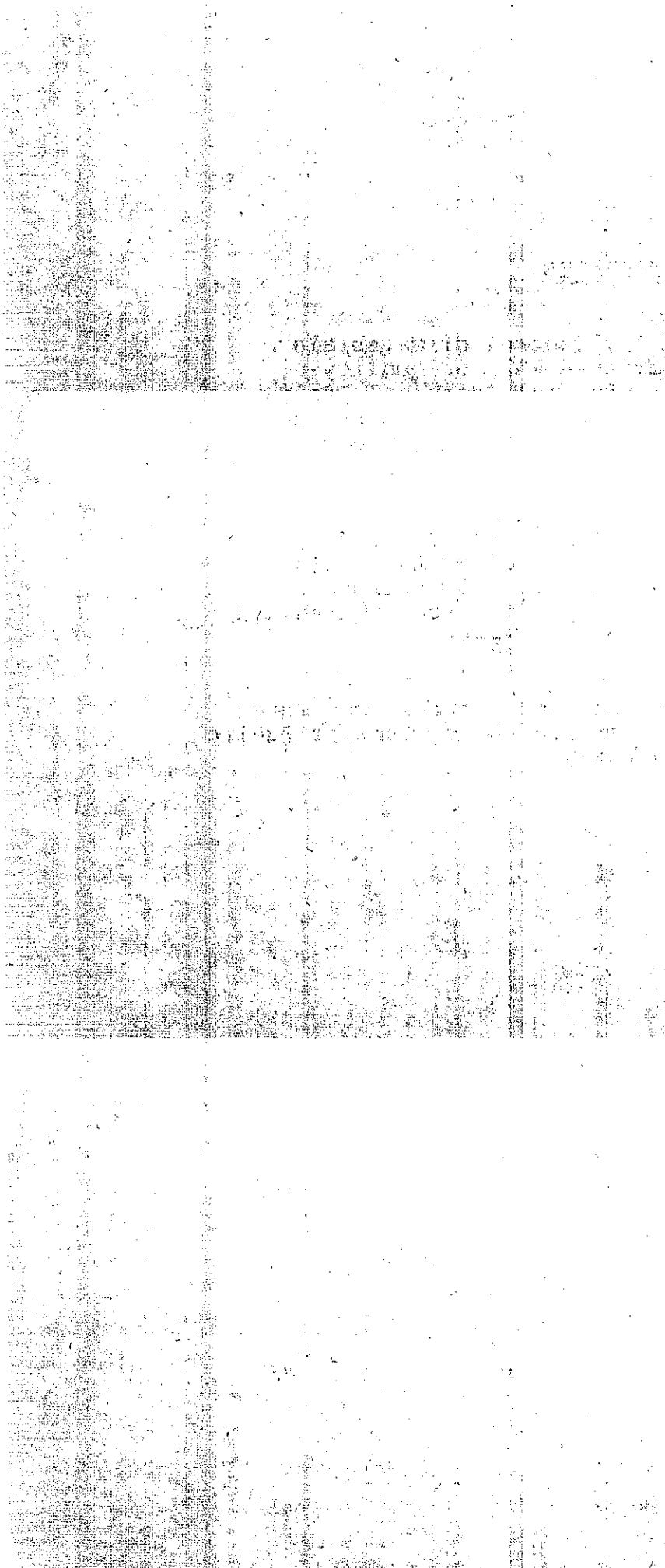


TABLE 1

A Comparison of some ASTM Specification Limits for Type II, Portland Cement with Routine Test Values Observed at the Materials and Research Department

	ASTM Specification Limit	Range of Routine Test Results, Matls. & Research Department, 1967
Compressive Strength, PSI		
3 days	1000, minimum	1590 - 3270
7 days	1800, minimum	2310 - 4690
Fineness Blaine Sq.Cm/Gm	2600, minimum (Individual test result) 2800, minimum (Average)	2880 - 4170
Loss on Ignition, %	3.0, maximum	0.4 - 2.2

TABLE 2

3-day Compressive Strength Results
by Brands for Type II, Modified Cements
Tested During 1965

Brand	No. of Samples	Average psi	Standard Deviation
1	47	2420	260
2	29	2600	246
3	36	2280	232
4	36	2490	256
5	14	2450	145
6	25	2380	254
7	18	2290	295
8	31	2180	222
9	15	2280	154
10	24	2420	288
11	34	2550	321
12	9	2110	177
13	23	2490	228
14	14	2490	159
15	40	2510	296
All	395	2400	310

TABLE 3

7-day Compressive Strength Results
by Brand for Type II, Modified Cements
During 1965

Brand	No. of Samples	Average psi	Standard Deviation
1	47	3520	328
2	29	3710	295
3	36	3270	273
4	36	3440	347
5	14	3470	234
6	24	3480	293
7	18	3280	371
8	31	3150	251
9	16	3310	215
10	23	3560	330
11	34	3530	390
12	9	3060	289
13	23	3640	293
14	14	3760	231
15	40	3540	362
A11	394	3460	390

TABLE 4

Fineness, Blaine, by Brand for
Type II, Modified Cements Tested
During 1965

Brand	No. of Samples	Average Fineness Sq.Cm/Gm	Standard Deviation
1	46	3407	153
2	47	3210	206
3	52	3490	197
4	47	3469	192
5	10	3500	121
6	31	3573	126
7	20	3730	181
8	46	3437	172
9	23	3724	160
10	35	3479	152
11	45	3552	224
12	13	3627	180
13	29	3474	173
14	31	3415	260
15	47	3459	158
All	522	3473	220

TABLE 5

Contraction in Air
Test Method No. Calif. 527,
By Brand for Type II, Modified
Cements Tested During 1965

Brand	No. of Samples	Average % Contraction	Standard Deviation
1	64	.0423	.0029
2	69	.0394	.0029
3	71	.0375	.0026
4	70	.0411	.0029
5	13	.0385	.0025
6	58	.0393	.0037
7	20	.0390	.0042
8	48	.0387	.0038
9	50	.0388	.0026
10	50	.0389	.0030
11	58	.0448	.0032
12	4	.0356	----
13	44	.0438	.0032
14	12	.0450	.0044
15	63	.0378	.0029
All	694	.0405	.0043

TABLE 6

Alkalies, Equivalent Na_2O by Brands
For Cements Tested During 1966

Brand	No. of Samples	Average % Na_2O	Standard Deviation
1	55	.505	.041
2	24	.517	.039
3	32	.503	.042
4	26	.536	.038
5	17	.403	.047
6	19	.354	.024
7	17	.448	.045
8	36	.463	.049
9	37	.555	.030
10	52	.255	.052
11	20	.449	.023
12	4	.378	----
13	32	.477	.047
14	12	.487	.044
15	27	.532	.042
All	410	.465	.101

Table 7

Ignition Loss by Brand for all Types Portland Cement Tested, 1964-1966

Brand	No. of Samples	Average Percent	Standard Deviation
1	93	1.26	0.183
2	98	1.14	0.161
3	145	1.07	0.182
4	80	1.33	0.192
5	15	0.94	0.145
6	64	0.91	0.232
7	39	1.06	0.253
8	78	1.04	0.223
9	59	0.94	0.193
10	81	1.29	0.258
11	68	1.14	0.158
12	16	1.42	0.208
13	54	1.10	0.180
14	24	1.32	0.164
15	97	1.21	0.231
All	1026	1.058	0.259

TABLE 8

A Comparison of Some Existing and Proposed Specifications for Type II Modified Portland Cement

Property	Present Specification Limits	Proposed Spec. Limit		Samples Failed in 1967				Number of Cement Mills Having Failures in 1967		Maximum Failure Rate of Any Cement Brand, %	
				Present Specs.		Proposed Specs.					
		Individual Test	Moving Avg. of 5 Tests	No.	%	No.	%	Present Specs.	Proposed Specs.	Present Specs.	Proposed Specs.
Compr. Strength, PSI											
at 3 days	1000 Min.	1600-3200	2000-2800	0	0	1	0.2	0	1	0	5.3
7 days	1800 "	2450-4450	3000-3900	0	0	0	0	0	0	0	0
Alkalies as Na_2O, %	.60 Max.	.60 Max.	.55 Max.	6	1.5	6	1.5	1	1	15.8	15.8
Fineness, Blaine Sq.Cm/Gm.	Individual 2600, Min. Average 2800, Min.	2800-4100	3100-3800	0	0	0	0	0	0	0	0
Contraction, %				15[a]	2.7	1	0.2	5	1	11.1	2.3
T.M. No. 527	.048 Max.	.051 Max.	.045 Max.	0[b]				0		0	
Loss on Ignition, %	3.0 Max.	2.0 Max.	1.5 Max.	0	0	1	0.2	0	1	0	2.2

[a] Strict interpretation

[b] Allowing .005% test tolerance

Table 9

Evaluation of 1967 Portland Cement Production Using Proposed Specification Limits and Control Charts

Property	Number Samples Tested	No. Samples Exceeding Spec. Limits	Number Tests[a] Waived	Number Rejections	No. Tests[a] Causing Moving Avg. to be Exceeded	Percent Failures	
						All Brands	Brand/W Highest Rate of Failures
Compressive Strength, psi							
at 3 days	444	3	2	1	7	0.2	5.3
at 7 days	430	0	0	0	5	0	0
Percent Alkalies as Na_2O	399	6	0	6	4	1.5	15.8
Fineness (Air Permeability)	482	0	0	0	4	0	0
Contraction, T.M. No. 527	545	2	1	1	2	0.2	2.3
Loss on Ign.	488	1	0	1	5	0.2	2.2

[a] Manufacturer warned when such incidents occur.